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Abstract

This paper reports on the difficulties of finding an accurate and consistent test method for quantifying the segregation of two fine powders. The background is from an industrial project investigating the feasibility of blending pulverised coal and crushed nickel ore in a bulk materials handling application, to ultimately produce nickel. The feasibility of combining the two products early in the process was investigated due to the blend requiring various different handling methods throughout the industrial process, making the constituent products susceptible to segregation. In the experimental investigation, representative samples of each bulk material were used to create a range of blends of the two products in the hopes of being able to develop a calibration curve to compare against collected samples. A number of tests were investigated; colour intensity, laser particle sizing, particle density, loose-poured bulk density, relative density and tapped bulk density. Each test method produced varied and at times inconsistent results and these will be discussed, however, ultimately one tapped bulk density method proved to show consistency.

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On the Difficulties of Quantifying the Segregation of Fine Powder Blends

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ABSTRACT This paper reports on the difficulties of finding an accurate and consistent test method for quantifying the segregation of two fine powders. The background is from an industrial project investigating the feasibility of blending pulverised coal and crushed nickel ore in a bulk materials handling application, to ultimately produce nickel. The feasibility of combining the two products early in the process was investigated due to the blend requiring various different handling methods throughout the industrial process, making the constituent products susceptible to segregation.

In the experimental investigation, representative samples of each bulk material were used to create a range of blends of the two products in the hopes of being able to develop a calibration curve to compare against collected samples. A number of tests were investigated; colour intensity, laser particle sizing, particle density, loose-poured bulk density, relative density and tapped bulk density. Each test method produced varied and at times inconsistent results and these will be discussed, however, ultimately one tapped bulk density method proved to show consistency.

1. INTRODUCTION

Many industrial applications handle bulk materials which have the potential to segregate and in the majority of those cases this is a phenomenon which is undesirable. The segregation of products or product blends occurs due to a number of factors such as; particle size, shape, density and friction. These factors influence the way in which particles interact and can result in the occurrence of segregation mechanisms such as; sifting (percolation), trajectory effects, avalanching and vibration [1-4].

There are many invasive and non-invasive methods employed for sampling blends to quantify the degree of segregation present, including, but not limited to; visual inspection [5] and image analysis [6-9], the use of sampling probes and sampling thieves [10-19], magnetic resonance imaging [20], near infrared (NIR) spectroscopy for real time monitoring of mixing (or lack thereof) [21, 22], acoustic emission [23] and high performance liquid chromatography (HPLC) [24].

With the focus of this paper being on powder blends, it was found that a large number of the published works investigating segregation of fine powders lie in the arena of pharmaceuticals, where it is of course critical that the correct dose of the active ingredient is combined with the larger proportion of excipient material [17, 24-26]. Boschetto [8] also provides a detailed summary of these and other methods, including their benefits and disadvantages.

Mixing or segregation trials have occurred in rotating drums [10, 14, 16] with the largest having a diameter of 152 mm, a v-blender [19] with a volume of approximately 15 litres or static tubes [11, 12]. The sizes of sampling probe and test apparatus presented are suitable for testing in the pharmaceutical industry or when evaluating sampling test methods, however, no specific literature was found dealing with the difficulties of sampling large industrial applications. The principles behind the sampling at the smaller scale are certainly valid, although the question of scale-up or at least the validity of the results after scaling up the procedure need to be carefully considered.

Of the range of methods employed to quantify the propensity for segregation (some of which are listed above), generally the non-intrusive methods would be more favoured, for example visual inspection/digital image analysis, as there is no physical disruption of the product being sampled. This process of image analysis, especially if there are a large quantity of images to be analysed spanning numerous tests, can become a monotonous task, and as stated by Agarwal [7], can affect one's motivation to generate sufficient data for statistical analyses to be completed.

For digital image analysis there is also a need for the individual products to be sufficiently different in colour to allow the analysis to accurately identify the constituents. In the situation where granular materials are being analysed, this is a relatively straight forward process because individual constituent particles are able to be identified, as has been shown previously by the author when determining the segregation of binary granular mixtures in storage bins [27, 28]. This is not the case when mixing extremely fine powders. With fine powder mixes, it is near impossible to distinguish one particle from another, regardless of which constituent is the focus of the analysis.

The focus of this paper is to explain the various methods employed in an attempt to produce a calibration curve from accurately determined proportions of the two fine powder products, from which experimental sampling could then be compared with certainty.

2. INDUSTRIAL PROCESS AND MATERIALS

In a recent industry project, the potential for segregation of a pulverised coal and crushed nickel ore was investigated. Both products were to be delivered to site in an ‘as received’ state from their respective mine sites and crushed in ball mills at an early stage in the process.

Samples of both pulverised coal and crushed nickel ore were supplied by the client, indicative of the size to be generated in the ball mills. Additionally, two blended samples were supplied based on site requirement; one containing 85% nickel ore by mass and one containing 95% nickel ore by mass, with the remaining proportion being pulverised coal. Table 1 details the particle and bulk properties for each of these products.

Table 1 Particle/bulk characteristics of the two bulk materials

	Pulverised coal	Crushed nickel ore
Loose-poured bulk density (kg/m ³)	532	725
Particle size, d ₁₀ (μm)	11.7	2.0
Particle size, d ₅₀ (μm)	51.7	20.5
Particle size, d ₉₀ (μm)	120.7	229.8
As received moisture content (%)	1.5	2.3

3. BENCH-SCALE EXPERIMENTAL CALIBRATION INVESTIGATION

Before any full scale segregation trials could be conducted, a method had to be developed that would allow the collected samples to be compared to known data, preferably via a calibration curve. The following sections explain the various methods trialled and the difficulties found in accurately and consistently developing a calibration curve. It was also acknowledged that in the full-scale segregation testing, specific samples would most likely have mixture quantities different to the four samples provided by the client. To allow this process, 21 blend samples were created. 200 gram samples were created by first stirring buckets of the constituent products to ensure homogeneity before taking representative sub-samples of each constituent to the required mass for each sample blend (5% increments by mass). To mix the constituents, the measured material was added to a plastic bag, sealed and then manipulated by hand for approximately one minute.

3.1 Mechanical Sieving

The first test method considered was mechanical sieving due to its general simplicity. However, due to the overlap in the particle size distribution of both products, as shown in Table 1, the analysis of the fine powder blends could not be achieved by mechanical sieving to separate the constituents. As a result, this method was disregarded. This method does have the potential to be viable when products have distinctly different particle size ranges.

3.2 Colour Analysis

The constituent products have distinctly different colours, the crushed nickel ore being orange/brown and pulverised coal being black and when a 50%/50% blend (by mass) is created, it naturally takes on a colour somewhere between the two extremes, as shown in Figure 1. Being that the constituent products have such a small particle size, the constituents are not readily identifiable as the colour looks to be uniform. The next step was to investigate whether producing blends in 10% increments (by mass) would allow a progressive colour variation that was able to be used to compare physical samples. When this was trialled, two main issues were observed. Firstly, as can be seen in Figure 1, the 100% and 50% crushed nickel ore images, although different in colour, are not sufficiently different that four additional 10% increments in between could be clearly identified. This was also the case for the four additional blends moving towards 100% pulverised coal. The second issue was the effect of variable lighting. Shadows or flash photography would regularly change the perceived colour of a sample. For these reasons, this method was abandoned.

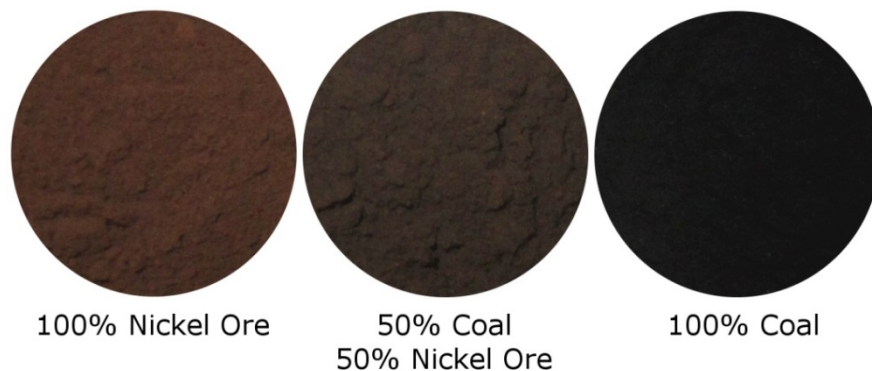


Figure 1 The colour of 100% crushed nickel ore, 50%/50% blend and 100% pulverised coal

3.3 Particle Size Distribution

The particle size distributions (PSD) of the four supplied product samples were analysed using a Malvern Mastersizer 2000 laser particle sizer on a wet basis, with an ultrasonic bath used to ensure there was no particle agglomeration during the tests. The results of the sizing tests are displayed in Figure 2. As can be seen, each sample has a peak value in the range 10 μm to 70 μm , which have an upward moving trend. On first inspection it was thought that this trend could be used for calibration purposes, however, on closer inspection it was found that the results of the 95% nickel ore / 5% coal blend was anomalous, in that the size distribution curve fell below the 100% nickel ore sample. Having 5% coal in this blend should have resulted in a curve positioned above the 100% nickel ore curve. The answer to why this occurred lies with additional information obtained from Figure 2. For the 100% ore sample and the 95% ore / 5% coal sample, there is a main size distribution curve and then there is an additional spike at approximately 1000 μm , indicating the presence of a small percentage of larger particles in each of these samples. A physical investigation of these two samples was performed by rubbing a small amount of material between thumb and forefinger. A small quantity of relatively larger particles could be felt, somewhat like the feeling of rubbing grains of sand between your fingers. It is assumed that this is a result of the original crushing process. It was assumed that even though small in quantity, these larger particles had enough of an effect to invalidate the potential creation of a calibration curve from this data – unless extensive screening was undertaken but this was not considered viable (or valid in the context of the project). Some further PSD tests were performed on other blend proportions (not shown in Figure 2), however, the results were scattered and the overall feasibility of using this method to produce a calibration curve was determined to be very low.

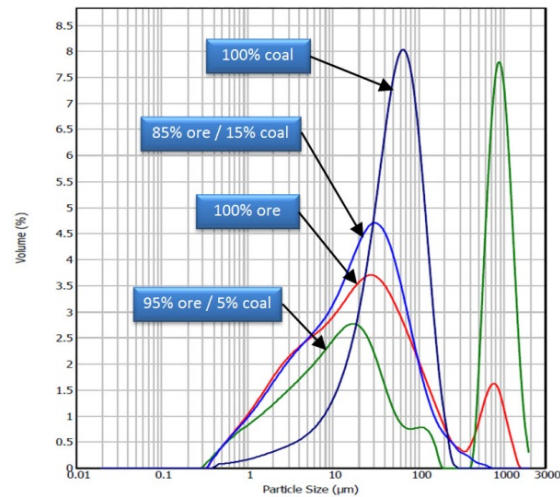


Figure 2 Particle size distributions of the four supplied products containing nickel ore and pulverised coal

3.4 Relative Density

A relative density test involves filling a graduated measuring cylinder with a known quantity of fluid, in this case water, and adding a known mass of a product. The product is then submerged in the fluid and the final volume measured. The mass divided by the change in volume provides the relative density of the product. This test method is straight forward and produced the data points plotted on Figure 3 for the four supplied samples. There was one major issue when conducting these tests. The test relies on the added bulk material being completely submerged in the water to allow a truly accurate volumetric measurement. What was noticed with both the crushed nickel ore and pulverised coal, as the sample was stirred into the water, was that the majority of the product did sink as expected from their bulk densities, however, due to the extremely fine particle size, some product would remain floating on the water surface and even stuck to the side of the measuring cylinder, see Figure 4. This meant that the results obtained were not accurate and further testing was not carried out.

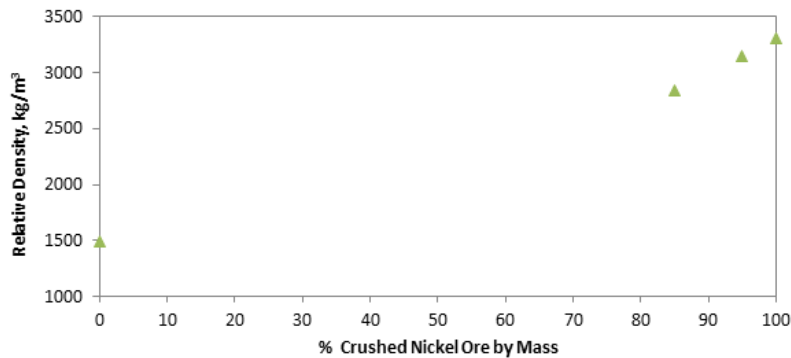


Figure 3 Attempts to produce a calibration curve for the nickel ore / coal blends using relative density



Figure 4 Example of pulverised coal sticking to the inside of the glass tube above the water layer

3.5 Particle Density

The particle density of a material is its mass divided by its true volume (excluding any voids). The mean particle density of product blends was determined using a manually operated stereopycnometer. Tests were carried out starting at 100% crushed nickel ore and in 10% incremental steps, by taking sub-samples from the 200g blends previously prepared. Care was taken to ensure the blend was mixed before the samples were taken. The results are shown in Figure 5, indicated by the red curve. Initially the trend was reducing in an expected way but then at the 60% crushed nickel ore blend, the results began to deviate. As can be seen, two tests were performed with similar results and then lower percentage tests were also varied. It was assumed that some segregation had occurred within the 200g sample even though care was taken in the extraction of the sample to be tested.

A second round of testing was then trialled, where exact measures of each constituent product were weighed and added directly to the stereopycnometer chamber. The results are again shown in Figure 5, this time represented by the blue curve. The results were looking promising down to the 40% crushed nickel ore blend and then there was a noticeable difference in results. As can be seen, there is a raised section in the curve which seemed anomalous. The closest curve fitting function that could be applied was a 6th order polynomial, represented by the black line on Figure 5. It is unknown what caused this raised section and repeat tests confirmed the original data. It is possible that the two constituent products have different porosity, allowing gas to penetrate into the products differently, which could result in invalid test results. However, no microscopic examination was conducted due to time constraints on the larger project. As a result of the variability in results seen in either method trialled, particle density was ruled out as a calibration method.

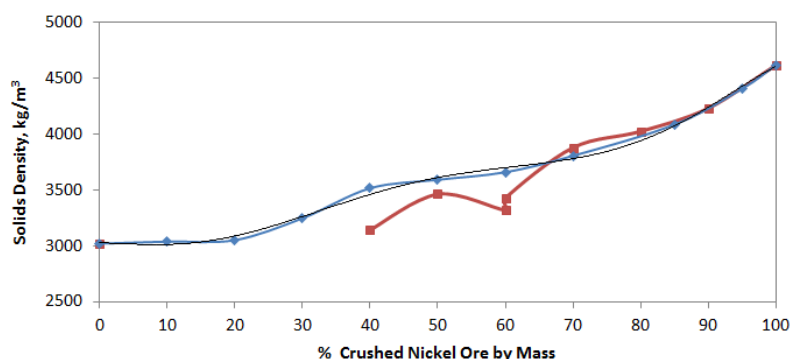


Figure 5 Attempts to produce a calibration curve for the nickel ore / coal blends using particle density

3.6 Loose-Poured Bulk Density

Representative samples of each supplied material were taken and carefully poured into a graduated measuring cylinder, ensuring minimal aeration or consolidation of the samples. The mass and volume of the sample were measured and from these two values, the density was determined from the “mass/volume” relationship. Multiple measurements were taken for each sample to ensure repeatable results were obtained. The averaged loose-poured bulk density results for the four supplied products are presented in Table 2 and Figure 6. It is evident from the four plotted data points that the loose-poured bulk density drops as the percentage of crushed nickel ore reduces, as is to be expected due to the pulverised coal having a lower loose-poured bulk density. One concern that arose during the measurement of the loose-poured bulk density was the fact that these two fine powders deaerated quite quickly and even a slight variation in volume when measuring, could have a dramatic effect on the resulting density value. Even though this density test is a commonly performed test for many bulk materials investigations, it was deemed to be potentially inconsistent due to the deaeration of these products.

3.7 Tapped Bulk Density

The tapped bulk density test is suited to products or blends which retain air, with the test removing air (deaerating) the product through vertical tapping (via a cam mechanism) of a graduated measuring cylinder containing the test sample. There are numerous standards for the determination of tapped bulk density; however, they have been developed for specific products in specific industries [29-33]. Each standard specified a different number of taps required from 100 through to 2500, but all had a common tap rate of 250 taps per minute.

The tapped bulk density of the four supplied samples was measured by running the test for a total of 100 taps. The calculation of the tapped bulk density is the same as for the loose-poured bulk density; however, the final volume of material is used in this case. Observations from the tests showed that when the percentage of coal increased in the sample, the more fluidic the material behaved as it was undergoing the tapping process. This resulted in a sample which was only partially aerated after 100 taps and on completion of the test the sample volume would continue to decrease for a short period of time (due to further natural deaeration). As a result, two measurements were taken: an instantaneous reading directly after the 100 taps and a settled reading once no more change in volume was observed (generally within the space of one to two additional minutes). Both sets of results are shown in Table 2 and Figure 6. As expected, the settled tapped bulk density had a higher value than the instantaneous tapped bulk density values. The results showed promise and also repeatability. The only concern was the limited number of taps used for each test.

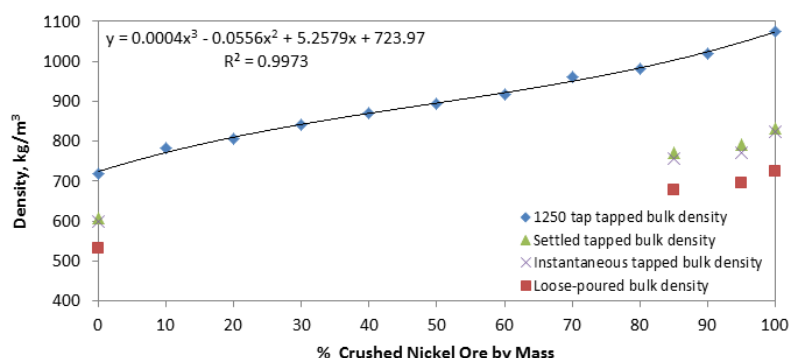
The deaeration effect seen in the tapped bulk density tests was not ideal as it would produce a degree of variability to the results obtained. The decision was made to apply a larger number of taps to ensure as much air as possible had been removed from the test samples, allowing for even more repeatability of results to be achieved. Referring again to the tapped bulk density standards mentioned previously, separate trials investigating number of taps were performed on a selection of the blends and it was found that after 1250 taps at a rate of 250 taps per minute, a steady-state value could be reached. The decision was made that this arrangement had the potential to produce an accurate and consistent calibration curve and so 11 tests were completed as shown in Table 3, to generate the data required. The resulting calibration curve can also be seen in Figure 6. It is obvious the effect of 1250 taps has had on the tapped bulk density results, producing much higher values. The data points are based on averaging of three repeat tests at each percentage increment. Applying a trendline allowed a 3rd order polynomial to be fitted with a high accuracy ($R^2 = 0.9973$). It was deemed unnecessary to use higher order polynomials as there was only a minimal increase in the R^2 values.

Table 2 Density values of the four products/blends

	Loose-Poured Bulk Density (kg/m ³)	Instantaneous Tapped Bulk Density (after 100 taps) (kg/m ³)	Settled Tapped Bulk Density (after 100 taps) (kg/m ³)	Tapped Bulk Density (after 1250 taps) (kg/m ³)
Pulverised coal	532	598	608	719
85% nickel ore / 15% coal	677	757	772	1002
95% nickel ore / 5% coal	696	770	791	1047
Nickel ore	725	823	832	1075

Table 3 Tapped bulk density using 1250 taps

Crushed Nickel Ore (percentage by mass)	100	90	80	70	60	50	40	30	20	10	0
Tapped Bulk Density (kg/m ³)	1075	1020	980	962	917	893	870	840	806	781	719

**Figure 6** Variation of density results for the product samples

This final calibration curve was ultimately used for the full-scale segregation trials, which have previously been detailed [34]. By analysing the tapped bulk density (1250 taps) of a segregation sample and applying it to the polynomial equation, the percentage of crushed nickel ore could be determined.

4. CONCLUSION

This paper has shown the difficulty in determining an accurate experimental test method for dealing with the measurement of segregation in fine bulk powder blends. In all test methods employed, variability of result was a common occurrence. The retention of air within the voids of the fine powders has been shown to create variability in the determination of the loose-poured bulk density and tapped bulk density. Only when the tapped bulk density test was performed over a substantial number of taps, 1250 taps being found suitable for these two fine powder products, was the tapped bulk density test able to generate consistent repeatable results which matched closely with the original blend constitution.

When measuring the particle density, it was found that taking a sub-sample of a larger sample produced quite variable results. This should be somewhat expected as there is every chance that there is already a degree of segregation present in the parent sample, regardless of how carefully it is handled. The solution to this would be to test the entire sample to ensure a complete picture of that sample is seen. This would not be feasible for the particle density tests in the stereopycnometer, due to the very small test sample used for each test and hence number of repeat tests required, but it is practical for the tapped bulk density test, which can use up to 250 ml of product in each test.

The overall findings showed that for these two fine powders and most likely other fine powders, the 1250 tap tapped bulk density test proved the most consistent, thus allowing the generation of a calibration curve. This was ultimately the method used to quantify the degree of segregation present in the full-scale testing performed as the overall end result of this investigation [34]. Further research is currently underway focussing on the tapped bulk density test, especially for products which fall outside those stipulated in the currently available standards.

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